

Carbonate cementing minerals in rhizoliths from Badain Jaran Desert: Implication for pedo-diagenesis and environment of dune soil

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1	Carbonate cementing minerals in rhizoliths from Badain						
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5	Qingfeng Sun ^{a,b,c*} , Kazem Zamanian ^d , Arnaud Huguet ^e , Guido L.B. Wiesenberg ^f , Tianlei Zhao ^g ,						
6	Ziqiang Lei ^a						
7							
8	^a Key Laboratory of Eco-function Polymer Materials of the Ministry of Education, Northwest Normal						
9	University, Lanzhou, Gansu, P.R. China						
10	^b State Key Laboratory of Palaeobiology and Stratigraphy, Nanjing Institute of Geology and						
11	Palaeontology, CAS, P.R. China						
12	^c Key Laboratory of Resource Environment and Sustainable Development of Oasis, Gansu Province,						
13	Northwest Normal University, Lanzhou, Gansu, P.R. China						
14	^d Institute of Soil Science, Leibniz University of Hannover, Herrenhäuser Str. 2, 30419 Hannover,						
15	Germany						
16	^e Sorbonne Université, CNRS, EPHE, PSL, UMR METIS, F-75005 Paris, France						
17	^f Department of Geography, University of Zurich, Zurich, Switzerland						
18	^g CAS Key Laboratory of Crust-Mantle Materials and Environments, School of Earth and Space						
19	Sciences, University of Science and Technology of China, Hefei, P.R. China.						
20							

21 Abstract

22 Pedogenic carbonates are a good proxy of soil formation and functioning because the morphology of minerals is directly related with the processes of 23 24 dissolution-precipitation. Carbonate rhizolith is one type of pedogenetic carbonate forming in modern soils of dunes and loess. The calcite micro-morphology with the 25 sedimentary hydrogeochemical conditions prevailing during their formation remains 26 open. Electron microscopy with an X-ray energy dispersive spectrometer and X-ray 27 28 diffraction were used to investigate in detail the micro-morphology of carbonate cements of the rhizoliths and obtain paleoenvironmental information in the dune soil 29 of Badain Jaran Desert in Northwest China. Two types of carbonates rhizoliths, 30 31 primary rhizoliths (PR) and reworked rhizoliths (RR), were found. The PRs are

32 present in situ within dune soils. The RRs are being eroded out from soil, potentially dislocated or transferred, and weathered at the soil surface. PRs displayed 33 34 homogeneous micritic mass of calcite crystals, with pores, cavities and voids among the mass. RRs displayed various morphologies of calcite crystals (polyhedral, 35 36 euhedral rhombic, tooth-like, prismatic and pillar), rosettes, short rods, and calcified fungal hyphae. The diagenesis differences at soil depth and surface affected the 37 38 crystal morphologies. The homogeneous micritic mass of calcite crystals of the PRs 39 were formed during the early diagenesis, preferentially in wet soils with semi-closed 40 and semi-redox conditions. In contrast, the cementing minerals of the RRs became bigger and clearer in the late diagenesis than during early diagenesis due to 41 42 recrystallization in open, dry and oxidative conditions at the soil surface. Therefore, the cement crystal morphologies of the rhizoliths can reflect the local environmental 43 and climatic conditions. These objects can be used as environmental and 44 pedo-diagenesis proxies in dune soil or sediments, recording the associated conditions 45 46 of moisture, temperature, wind, erosion, weathering, solar radiation at the soil surface 47 or at depth. Therefore, assessing the micromorphology of pedogenic carbonates has important implications for soil ecology and evolution as well as plant root physiology 48 49 in deserts.

50

51 Keywords

52 Carbonate minerals; Scanning electron microscopy; Energy dispersive X-ray
 53 spectroscopy; X-ray diffraction; Crystal morphology; Soil diagenesis environment
 54

55 **1.Introduction**

56 Calcium carbonate (CaCO₃) mostly exists in three crystalline forms, which are, 57 in the order of stability, calcite, aragonite and vaterite (e.g., Gopi et al., 2013; Sekkal 58 & Zaoui, 2013; Addadi & Weine.,1997; Simkiss, 1964). Vaterite crystals have a 59 hexagonal structure, and aragonite crystals show an orthorhombic structure, while

calcite crystals present a rhombohedral structure (Liendo et al., 2022; Jimoh et al., 60 2018). Investigating the micro-morphology of calcium carbonate in soils, sediments 61 62 and rocks can help in reconstructing past environmental conditions (Alonso-Zarza, 2018; Verrecchia & Verrecchia, 1994; Monger et al., 1991) The occurrence and 63 morphology of pedogenic carbonates, including size, shape and distribution are 64 valuable indices of pedo-environments and pedogenic processes (Golubtsov et al., 65 66 2021; Nyachoti et al., 2017; Durand et al., 2018; Diaz-Hernandez et al., 2018; Milliere et al., 2018; Eren et al., 2018). 67

68 Calcium carbonate found specially in rhizosphere microecosystems shows a unique relationship with several factors such as organic matter, micro-organism and 69 plant distribution, as well as moisture and leaching processes (Huguet et al., 2020; 70 Casado et al., 2014; Becze-Deák et al., 1997). Carbonate micro-morphology includes 71 calcified root cells (Microcodium), CaCO₃ hypocoatings, needle fiber calcite, 72 73 powdery calcite coatings, and calcite pseudomorphs on gypsum (Golubtsov et al., 2021; Kosir, 2004; Becze-Deák et al., 1997). However, despite many decades of 74 75 thorough descriptive and interpretative work of these minerals, relating calcite 76 micro-morphology with the predominant sedimentary hydrogeochemical conditions 77 during their formation remains a challenge. Environmental interpretations of the calcite micro-morphology have been hampered by the fact that the calcite 78 79 morphogenesis results from complex interactions between different physico-chemical parameters which often act simultaneously (e.g., carbonate mineral supersaturation, 80 Mg/Ca ratio of the parental fluid, organic and inorganic additives, Mercedes-Martín et 81 82 al., 2021).

The micro-morphology of calcium carbonate within carbonate rhizoliths, such as calcite rhomb chains, isolated calcite rhombs, calcified spherical bodies, calcified epilithic and chasmolithic filaments, grain-coating calcite needle mats, is related to their formation characteristics (Jones & Ng, 1988). Since the factors controlling the morphology of calcite crystals are not fully understood, it is necessary to rely on other materials to obtain an insight into this aspect of the problem. There is probably no

89 universal cementation scheme for rhizoliths, the characteristics of the cements being a function of the local conditions. The nature of the plants themselves may play an 90 extremely important role in the cementation of rhizosphere (Sun et al., 2021, 2020; 91 Marasco et al., 2018) or within roots (Huguet et al., 2021; Luo et al., 2021; Gocke et 92 al., 2014). Plants were shown to have an obvious effect on the diagenesis of 93 carbonates in vadose environments (Sun et al., 2021, 2020; Marasco et al., 2018; 94 Jones & Ng, 1988). Nevertheless, the characteristics of the cements can be controlled 95 to a large extent by the activity of the microorganisms that are associated with the 96 plant roots. 97

A large number of studies on rhizoliths has been published over the last decades, 98 but detailed investigations of the microscopic morphology of calcium carbonate 99 within carbonate rhizoliths remain scarce, with the description of microscopic 100 morphology of calcium carbonate included in some case studies (e. g., Sun et al., 2021, 101 2020, 2019a, b,c; Gao et al., 2020a, b; Huguet et al., 2020; Li et al., 2020, 2017, 102 2015a, b; Zhu et al., 2019; Alonso-Zarza, 2018; Barta, 2011; Klappa, 1980). 103 104 Nevertheless, pedogenic carbonates are a good promising proxy of soil formation and 105 functioning because the morphology of minerals is directly related with the processes of dissolution-precipitation. Thus, they can provide information on climate and water 106 regime dynamics during or even after the lifetime of plants, whose roots entail 107 108 formation of these structures. Electron microscopy with an X-ray energy dispersive spectrometer (SEM/EDS) was shown to be a powerful tool to investigate in detail the 109 micro-morphology of carbonate cements and obtain paleoenvironmental information 110 (Kuznetsova & Khokhlova, 2012). 111

Apart from many other known areas worldwide, carbonate rhizoliths of the Alashan Deserts of Northwest China have been intensively studied over the recent years using different tools (lipid biomarkers, ¹⁴C dating, major and trace elements, phytoliths, cementing mineral morphology, carbon and oxygen isotopes), allowing the historical analysis of climate change (Sun et al., 2021, 2020, 2019a, b; Li et al., 2020, 2017, 2015a, b; Gao et al., 2020a, b; Zhu et al., 2019). However, only few information

on rhizolith genesis were obtained. Several key issues remain open, such as the age 118 and formation mechanisms of the rhizoliths, including the influence of the vegetation 119 type and soil microbial diversity on the formation of these objects (Sun et al., 2021, 120 2020). For instance, the rhizolith ages remain often unclear due to the age 121 122 discrepancies between their carbonate cementing minerals and the inner root relicts (Sun et al., 2019a). The influence of climate change on the root tubes (Li et al., 2015) 123 124 has still to be investigated. This is even further complicated by the fact that the rhizoliths are often not found in intact relationship with their forming plants. The 125 identification of traces of life based on rhizolith characterization is usually 126 challenging, especially in very old rocks, as the morphological and chemical 127 signatures of biominerals and microfossils are subtle, of microscopic size and 128 inevitably altered with aging. Nevertheless, the rhizoliths of the Alashan Deserts are 129 of special interest, as they were shown to form only around the dead roots of 130 Artemisia and to save the complete information of mineralization of calcium 131 132 carbonate during Holocene in modern dune soils through pedodiagenetic effects (Sun 133 et al., 2021, 2020). Therefore, this work aimed at investigating the micro-morphology of calcium carbonate of the rhizoliths occurring in dune soils from Badain Jaran 134 Desert in the Alashan Deserts. Such a study was performed using SEM, EDX and 135 XRD. The cement micro-characteristics of the carbonate rhizolith were related to the 136 environmental conditions and processes taking place in and on the soils, allowing to 137 better constrain the conditions of these objects as environmental proxies. This study 138 provides a reference and model for future rhizolith studies of deserts worldwide. 139

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141 **2. Study area**

Badain Jaran Desert (BJD) (39°04'15"N to 42°12'23"N, 99°23'18"E to 143 104°34'02"E) is in North-western China (Fig. 1a, b), covering an area of 144 approximately 52,100 km². It is subjected to a transitional climate between the Asian 145 summer monsoon and westerly belt (Wang F., et al., 2015). The elevations of the BJD

range between 1,000 and 1,500 m a.s.l. and decrease from southeast to northwest (Zhu
et al., 2010). This desert comprises the tallest mega-dunes the world, generally
200–300 m high and 3-5 km long, with lakes frequently occurring between the dunes.
The latter are especially dense in the south-eastern portion, where they can reach up to
500 m a.s.l. Towards the northwest alluvial/lacustrine plain, the size of sand dunes
progressively decreases and the lakes gradually disappear (Fig. 1, 2).

152 The mean annual precipitation is currently 40-120 mm, falling mainly in summer. The mean annual potential evaporation is over 2500 mm. The conventional 153 precipitation (below 5 mm) accounts for approximately 90% of rainfall events (Wang 154 et al., 2013). The soil heat flux shows the largest diurnal variations at 5 cm depth and 155 only slight variation at 20 cm depth (Li et al., 2012). The mean annual air temperature 156 ranges from 9.8 to 10.2°C and the mean annual wind speed varies from 2.8 to 4.6 m/s. 157 The prevailing wind direction over the year is northwest, related to the winter 158 monsoon from the Siberian High-Pressure System (Zhang et al., 2017). 159

The vegetation of this desert is dominated by shrubs and subshrubs, including *Artemisia, Agriophyllum, Triglochin, Glaux, Aeluropus, Haloxylon, Nitraria, Tamarix,*and *Reaumuria* species (Wang M. et al., 2015; Yang et al., 2003).

The dune soil sand sources are the weathered debris and sediments of the 163 northwest Gobi and the alluvial fan of the Heihe river. The northwest winds and 164 windstorms have transported them to the desert (Fig. 1. Dong et al., 2013, 2009). The 165 grain size of surface sand is dominated by well-sorted, rounded, pinky-grey, middle to 166 fine sand (of feldspar and quartz), containing only slight amount of melanocratic 167 minerals (such as hornblende, pyroxene and magnetite). Carbonate minerals are in 168 low abundance in the silicate desert sands (Bai, 2011; Gates et al., 2008). The average 169 carbonate content in soils was reported as 4.6 % by Wang et al. (2004) and 6.5 % by 170 Sun et al. (2019a). 171

The thickness of the upper dry layer of the mega-dunes in this desert is only about 20-30 cm, with a conspicuous wet layer under the upper dry layer. Between 0.2-1.5 m depth, the water content is usually 1-5% (wt, Wang et al., 2019; Zhao et al.,

175 2011; Gu et al., 2004) and is up to 10% at 90 cm depth with well-preserved sand bedding (Liu et al., 2016). At 2 m depth, water content is about 65% (pore saturation; 176 Zhao et al., 2011). Water in the sandy soil layer is mainly divided into adsorbed water 177 and gravity water. Adsorbed water exists in the form of a water film and is thus 178 179 termed film water. It is generally thought that if the water content of the sand layer is <3% then it is adsorbed water, and if the water content is >3% then it is gravity water. 180 Adsorbed water, which is derived from rainfall, moves slowly downwards and is 181 generally a minor contribution to groundwater (Zhao et al., 2017). In contrast, gravity 182 water moves rapidly downwards within a soil and is the most important supplement of 183 groundwater in depressions and lakes. Gravity water with a content above 5% (wt) 184 185 was found in some sand layers of the BJD (Zhao et al., 2017). The precipitation in this desert plays an important role by providing water to the soils and lakes (Ma et al., 186 2019; Yue et al., 2019). However, in somewhere in the hinterland of the desert, the 187 soil water contents are uneven and variable from 0.36-2.98% along depth changes 188 189 from 4.3-9.7m, due to micro-geomorphologies differences (Dong et al., 2022; Zhao et 190 al., 2017). Soil water content is less in the surface soil because it suffers stronger evaporation, while the deep soil water (> 200 cm) is hardly influenced by the 191 evaporation (Jin et al., 2021). 192

193

194 **3. Methods**

Because of the vast sand sea area, poor accessibility and the dominance of an 195 extremely arid climate, field explorations were only conducted in zones located in the 196 197 south-eastern part of the desert, where many mega-dunes, lakes and low small dunes next to the desert margin are present (Fig. 1, 2). In the exploration area in the 198 hinterland of the desert, rhizoliths were only found at six sites (Fig. 2, red points A-F). 199 Detailed survey and examinations for landscape, vegetation, modern root, 200 sediment/soil, rhizolith exposure and morphology were carried out around these sites. 201 202 At each site the most representative samples of carbonate rhizoliths and their typical

- features were intensively described and photographed. Typical rhizolith samples werecollected for laboratory analyses and further photographing (Sun et al., 2021).
 - 205 Care was taken to keep the rhizoliths intact for detailed description of their characteristics in the field down to the ultramicroscopic observations in the laboratory. 206 207 Samples were chosen based on their typical external characteristics for determining scanning electronic microscopy. Energy dispersive X-ray 208 spectroscopy, 209 micro-morphological texture and cement mineralogy analyses by microscopy, as well 210 as cathodoluminescence and staining were all performed at the Institute of Northwest 211 Petroleum Geology, Lanzhou, China (Sun et al., 2021). The micro-scale morphology and chemical compositions of the rhizolith cement were determined by scanning 212 electronic microscopy (SEM; FEI Quanta 450) coupled with Energy dispersive X-ray 213 spectroscopy (EDS). Fragments of the rhizoliths were gold coated, and then analysed 214 by SEM coupled with EDX, as previously described (Sun et al., 2021). Powder X-ray 215 216 diffraction (XRD) was recorded on a Japan Rigaku Smart Lab X-ray diffractometer 217 using Cu-Ka irradiation (λ = 0.154056 nm) to determine the mineral phase, and the 218 sample was scanned from 10 to $70^{\circ} 2\theta$ and at a step of 0.02° , at CAS Key Laboratory of Crust-Mantle Materials and Environments, School of Earth and Space Sciences, 219 Science and Technology of China, Hefei. China. 220 University of The 221 micro-morphological terms of carbonate minerals crystals are employed based on Mercedes-Martín et al. (2021)and Minerals.net 222 (https://www.minerals.net/mineral/calcite.aspx). 223
 - 224

225 **4. Results**

4.1. Field features of rhizoliths

Two types of rhizoliths were distinguished based on the outcrop states: reworked (RR) and primary rhizoliths (PR). The RRs are being eroded out from soil, potentially translocated and weathered on the soil surface under solar radiation and wind erosion, and they are thinner and harder than the PRs. In contrast, the PRs are present within dune soils and keep their original characteristics *in situ*, without being eroded,transferred and weathered.

233 4.1.1 RRs on soil surface

234 RRs were only found in Sites A, B and C. Their general features are described235 below.

At site A (Sumujilin Lake, 39°48'N, 102°26'E), the rhizolith outcrops were found at the feet of the windward gentle slopes of the mega-dunes situated above the east of the Sumujilin Lake (Fig. 3a). Brown purple RRs were present as fragments eroded out of the sand. The fragments of typical rhizoliths with bright dark brown colour and rills are hard and varnished brightly. Their centres are hollow (Fig. 3b).

At Site B (Nuoertu Lake, 39°46'N, 102°27'E), rhizolith outcrops were found along the gentle slopes of the mega-dunes on the east side of the Nuoertu Lake. RRs occurred as enormous fragments as at Site A. However, the fragments were white, corresponding to the inner parts of the central hollows of former roots subjected to weathering. In some rhizoliths, relicts of fine root hairs were observed. Weathered rills were apparent on the outer surfaces of the big tubes. The small broken pieces of rhizoliths were white and fragile due to weathering (Fig. 4).

At Site C (Baoritaolegai Lakes, 39°35'N, 102°28'E), rhizoliths were also found along a windward gentle slope between two mega-dunes, south of Baoritaolegai Lakes. RRs were found as broken tubes. Their general features are similar to the above sites (photos were omitted).

4.1.2 Vertical RRs above soil and PR features within soil

Both PR and RR were found at Sites D, E, F. But the RRs are vertical above soil and they are the upper parts of the PR underground.

At Site D (6.0 km east to the Gate of the BJD National Geological Park, 39°27'N, 102°26'E), RRs fragments are lying intricately at the sand surface. However, only a few of the RRs were found standing vertically above soil. PRs are present underground, below the vertical standing rhizoliths above soil surface. The upper part above ground is thinner and harder than the underground primary part within soil because of weathering and erosion (Fig. 5a). Once broken, they display decayed root
relicts within the central hollowness, surrounded by a white hale carbonate cement
(Fig. 5b). After having been dug out for some time, they became harder quickly than
before due to losing soil water at the site.

At Site E (39°25'14.56"N,102°8'55.25"E), their general features are similar to those of Site D. After having been dug out, the PR show brown interior with preserved root relicts when broken (Fig. 6). PR are soft, while RR are hard.

At Site F (39°29′57″N,102°12′48″E), their general features are similar to those of Site D. PR were broken easily by gentle hand-force. The centre of the decayed root relicts is brown (photos were omitted).

4.2. SEM features and EDS mineral compositions of rhizolith cement

271 Ten rhizolith specimens – one or two from each site - were chosen for laboratory
272 analyses, with determination of micro-morphological texture and mineral composition.
273 Their SEM features differ from each other and are detailed below.

274 4.2.1 RRs on soil surface

275 Site A

The cement of the RR is tight and densely compact around the sand particles (Fig. 276 7a). Some of the cementing minerals show polyhedral crystals and many pores occur 277 within the cement (Fig. 7b). They also display bigger euhedral rhombic crystals, or 278 tooth-like crystals around the walls of the pores, against the dense fine crystal cement 279 background (Fig. 7c). Moreover, they present both aragonite rosette morphology (Fig. 280 7d) and equant polyhedral crystals characterized by well-developed crystal faces and 281 282 slightly smooth edges and corners (Fig. 7e). Generally, all EDS spectra reveal that the cements are calcium carbonate (Fig. 7f). 283

284 Site B

The cement of the RR is compactly tight and dense around sand particles (Fig 8a). The morphology of the cementing minerals shows polyhedral (Fig. 8b) and short rods crystals (Fig. 8c). Their mineral compositions are calcite with a little of Mg (Fig.
8d) based on EDS analysis.

289 Site C

The cement of the RR is not as tight and densely compacted around sand particles as it was at sites A and B. However, the cement shows short rods shape (Fig. 9a) and calcified fungal hyphae-like networks with many pores, cavities and voids (Fig. 9b). The cement is calcium carbonate, as indicated by EDS (spectrum is omitted).

- 295 4.2.2 Vertical RRs and PRs
- 296 Site D

The rhizoliths are constituted of two parts, the upper parts-RR at the soil surface and the lower parts-PR within soil. The RR cement shows short rods crystals (photo is omitted). The PR either displays homogeneous micritic mass (Fig. 10a) with a lot of pores, cavities and voids (Fig. 10b), or dense prism/pillar-like calcite crystals (Fig. 10c). The cement is mainly constituted of calcium carbonate with only low amounts of Mg (EDS spectrum is omitted).

303

Site E

At this site only one PR was analysed. The lab tests of upper RRs were done. The cementing minerals are homogeneous micritic mass, which are growing along the surfaces of sand particles (Fig. 11a). The calcite crystals form thinner layers without obvious and clear crystal shapes and not thick enough to fill the pores among the particles (Fig. 11b, c). Based on EDS analysis, the cement composition is calcium carbonate. (Fig. 11d).

310 Site F

Similar to site D, the cement of the RRs above soil surface is made of short rods
crystals (photo is omitted). The PR within soil display homogeneous micritic mass
(Fig. 12), with pores, cavities and voids (photo is omitted). The PR cement is mainly

constituted of calcium carbonate with low amounts of Mg and NaCl (EDS spectrum isomitted).

The micro-morphologies and compositions of the cementing minerals and their diagenesis features are summarised in the Fig. 13. The micro-morphologies show the following key features: (i) the PRs have homogeneous micritic mass, with pores, cavities and voids; (ii) the RRs on or above soil surface have big and clear (polyhedral, euhedral, rhombic, prismatic, rosette) crystal shapes and short rods, with occasional hair-like, or fungi-form crystals; (iii) vertical upper RRs of the PR have only short rods; (iv) the cement minerals compositions is calcium carbonate with a little of Mg.

4.3. XRD mineral compositions of rhizolith cement

324 XRD results confirmed that the carbonate cements are all calcite (Fig. 14). In 325 addition, sand particles like quartz and plagioclase feldspars (matched well with 326 standard card of anorthite and anorthite, sodium) were also detected. This is consistent 327 with section cathodoluminescence observations (Sun et al., 2021).

At sites A, C and F, SEM showed that aragonite is also present by its shape in the cement. However, no diffraction peaks of aragonite were detected in the corresponding XRD results. Therefore, the content of aragonite is likely too low to be detected by XRD (Fig. 14a, c, f).

332

333

334 **5. Discussion**

5.1 General SEM characteristics of the cement of the PRs and biogeochemical implications

337 5.1.1 Early diagenesis features within soil

338 Within the underground dune soil, the diagenesis environment of primary 339 rhizoliths is characterized by weak redox conditions (due to semi-closed conditions) 340 with comparatively higher moisture content, and lower stable temperature than those at the soil surface. Therefore, the conditions of the early diagenesis process arecomparatively steady in deep soils.

Within soil, vascular plants and symbionts increase chemical weathering of the 343 silicates, which results in increasing of Ca^{2+} and HCO_3^{-} availability and pedogenetic 344 carbonate production (Brasier, 2011). There are two Ca²⁺ sources for rhizolith cement: 345 1) authigenic carbonates from in situ chemical weathering of soil primary silicate 346 minerals such as feldspar, mica, amphibole and pyroxene; and 2) dissolution of in situ 347 primary lithogenic carbonate rock debris such as limestone and dolomite (Sun et al., 348 2019a). Moreover, dead root decomposition leads to increased CO_2 and HCO_3^{-1} 349 production around the rhizosphere (Sun et al., 2021,2020). Then dry periods increase 350 351 the calcium concentration in the soil solution from which $CaCO_3$ precipitates by the evapotranspiration and release of CO_2 with temperature rise (Sun et al., 2022, 352 2021,2020). Precipitation is also associated with the dissolution and recrystallization 353 of carbonate taking place in the parent matrix, such as soil, sediment and rocks (Eren 354 355 et al., 2008). In the dune soil of this desert, the semi-closed environment is favourable 356 to both root organic decay and formation of HCO_3^- solutions among the pores of the sandy soil. However, the balance between root decay-CO₂-HCO₃ and calcite growth 357 is the key point making the soil micro-environment around the dead roots favourable 358 to the aforementioned process. Such a balance is controlled by factors like soil 359 moisture, soil depth, wind, as well as precipitation or/and temperatures. Such 360 parameters could not be measured in the field but would have been useful to better 361 362 understand the details of rhizolith diagenesis.

363 5.1.2 Origin and implications of cement crystal micro-morphology

Under the aforementioned diagenesis conditions, the cementing minerals of the primary rhizoliths display (i) homogeneous micritic mass in thin layers without obvious and clear crystal shapes, precipitating mainly on particles surface (Fig. 10a; 11a, b, c;12); (ii) prism/pillar-like calcite (Fig. 10c); and occasionally (iii) pores, 368 cavities and voids among the micritic mass (Fig. 10b). Their origins and implications369 are explained as follows.

The presence of homogeneous micritic mass implies that the fine calcite crystals were in their initial nucleation and quickly precipitated at the beginning of diagenesis, which cannot be observed even under high magnification (Zhou & Chafetz, 2009; Alonso et al., 2004).

374 The prismatic/pillar-like calcite crystals were subjected to diagenesis for a longer time than homogeneous micritic mass, as shown by the larger size of the former. This 375 suggests that the supersaturation degree of mineral solution changed during the 376 mineralization. During the formation and precipitation of crystals, the solution 377 378 became supersaturated, allowing the formation of the crystal nucleus. Then, the diffusion and precipitation of ions at the surface of the crystal nucleus led to the 379 crystal nucleus growth and later to the formation of crystal particles and even big 380 crystals when concentrated. If the supersaturation degree is too high, the reaction of 381 Ca^{2+} and CO_{2-3} will be too quick to control the crystal morphology. In contrast, when 382 383 the supersaturation degree is low, the growth velocity of crystal is low, letting enough 384 time to the formation of the surficial crystal (Cheng et al., 2014). An experiment investigating the precipitation of non-marine calcite confirmed that low calcite 385 supersaturation conditions are prone to form single-crystal prismatic to di-pyramid 386 387 calcite precipitates (Mercedes-Martin et al., 2021). This suggests that homogeneous micritic mass cement formed rapidly, with enough CO₂ supplied by root decay, and 388 389 that the short rods calcite crystals formed in low concentration of mineral solutions 390 and/or were subjected to secondary diagenesis after having been formed.

The pores, cavities and voids in calcite cement imply that microorganisms and CO₂ gas played key roles in calcium carbonate crystallization through fermentation effect. This is supported by the following evidences from previous studies: (i) These characteristics are similar to those of the carbonate crystals in Mu Us Desert in northern China, with a rough and porous appearance caused by bacterial strains (Liu et al., 2018). Therefore, the pores, cavities and voids in the BJD rhizoliths might be 397 caused by microorganisms. (ii) Irregular carbonate crystals with broken faces and holes on surfaces were suggested to be formed in humid conditions (Kuznetsova & 398 Khokhlova, 2012), which could be the case in the BJD. (iii) Increasing contents in 399 (alginic) acid were shown to contribute to the formation of curved and poorly faceted 400 401 crystal morphology with higher sphericity (Mercedes-Martin et al., 2021). This means that the pores, cavities and voids of the BJD rhizoliths could have been formed in an 402 403 acidic microenvironment of soil. The release of CO₂ from Artemisia root decay could 404 have led to the formation of such an acidic environment with gas bubbles (Sun et al., 2020). 405

These hypotheses are in agreement with the diagenesis features within deep dune 406 soil and with the presence of microorganisms like fungi in the pores among sand 407 particles. In fact, roots and associated microorganisms play a very important role for 408 the pedogenic carbonate formation, not only around living roots (Eren et al., 2018), 409 but also around dead roots (Sun et al., 2021,2020). For instance, microbialites have 410 411 been found in desert soils and tropical forest soils as well. Myxococcus, usually found 412 in soils rich in organic matter and decaying material, can precipitate calcium 413 carbonate independently of being metabolically active or killed, though different polymorphs of calcium carbonate can be formed (Zhu & Maria, 2016). Root decay 414 under microbial decomposition induce gases bubbles in mineral solution and produce 415 416 calcite with pores, cavities and voids through different metabolic pathways. Most microbial cells provide nucleation sites for carbonate formation, with appearance of 417 microbial textures like cavities, voids, fibric and hair-like shape. Previous studies also 418 419 confirmed that carbonate precipitation can be caused by soil microorganisms 420 (Khormali et al., 2020; Menezes et al., 2019; Wang et al., 2016; Zamanian et al., 2016; Vepraskas, 2015). 421

However, there is still an ongoing debate questioning whether the organisms were alive during the calcification (Eren et al., 2018). Where carbon comes from and whether atmospheric CO_2 can be deposited via this biomineralization process remain unknown, representing an important gap in the knowledge of carbon sequestration via

426 microbial carbonate deposition (Liu et al., 2018). In this case, organisms may use root remains as the source of carbon for biomass growth, resulting in increased 427 concentrations of calcium along the dead roots, with fungi digesting carbon 428 acquisition, leading to CaCO₃ precipitation. This process can take place both at the 429 surface or belowground. It is unclear whether microorganisms act like a catalyst by 430 increasing the medium pH, leading to the saturation of soil solutions and causing 431 432 calcite precipitation (Barta, 2011). Physicochemical and biogenic processes may have worked together to form variant crystalline forms (Verrecchia & Verrecchia, 1994). 433 Overall, several factors should be further investigated in this desert: (i) the microbial 434 diversity in the bulk soil, both in living and dead rhizosphere; (ii) the microbial 435 influence on calcite formation; (iii) a better understanding of why rhizoliths are 436 formed only around the dead Artemisia roots. 437

438 5.2 General SEM characteristics of the cement of RRs and

439

biogeochemical implications

440 5.2.1. Late diagenesis features at soil surface

The Badain Jaren Desert is characterized by large thermal (day-night) amplitude 441 and seasonality, strong wind and little precipitation (cf 2. Study area). The 442 temperature at the desert surface soil can reach up to 70-80°C. Night temperatures in 443 deserts are typically much cooler than daytime temperatures (Khormali & Monger, 444 2020). This heat is transferred into the soil profile downward, with a gradient of about 445 446 0.5° C per 10 cm until the temperature reaches the approximate mean annual air temperature at a depth of ca. 50 cm (Soil Survey Staff, 1999). Simultaneously, the 447 high-low temperature changes, extreme radiation and strong winds can enhance the 448 aridity of the surface soil, resulting in an increase of the mineral concentration in the 449 soil particle pores. Harsher conditions for microbial growth can be found in shallow 450 desert surface soils vs. deep ones, as only microorganisms resisting to high 451 temperatures can survive (Monger et al., 1991). The RRs are subjected to strong 452 aridity, high temperature changes, solar radiation, and furious wind at the dune 453

454 surface. Comparatively, the lying RRs on the soil surface are older than the vertical455 ones which are the upper parts of the PRs above ground.

456 **5.2.2** Origin and implications of cement crystal micro-morphology

The cements of the RRs show the following characteristics: (i) the horizontal lying RRs: polyhedral crystals of calcite with well-developed crystal faces and slightly smooth edges and corners (Fig. 7b, e; 8a, b), or prismatic crystals (Fig. 7c; 8c), rosette shapes (Fig. 7d), calcified fungal hyphae networks (hair-like, or fungi-form crystals (Fig. 9b); (ii) the vertical RRs: short rods of aragonite (Fig. 9a). This implies the following regarding the origin and diagenesis conditions of the RRs:

As shown in Fig. 13, homogeneous micritic mass recrystallized to short rods and 463 464 the short rods recrystallized to big clear (polyhedral, euhedral, rhombic, prismatic, rosette) crystals. Because the vertical RRs were eroded out from PRs present in soil, 465 the formation of these short rods calcite crystals at the dune soil surface was subjected 466 to a different diagenesis from the homogeneous micritic mass of PRs present 467 468 underground. Recrystallization of homogeneous micritic mass of RRs took place after erosion and weathering due to the loss of soil water and the high temperature near the 469 soil surface. Carbonate accumulations were formed in dry periods and in quiet 470 conditions from solutions of normal concentration (Kuznetsova & Khokhlova, 2012). 471 This is in line with a quick alternation of day-night temperatures (with fog some time) 472 and short shower events in the desert, providing water for dune soil in depth and 473 causing chemical weathering of soil minerals like feldspars and root decaying. 474 Carbonate mineral solution forms in the soil. Then, strong alternating wet and dry 475 476 conditions (due to strong wind, solar radiation and high temperature) promote and enhance the dissolution and precipitation of CaCO₃ and consequently formation of 477 secondary carbonates (Becze-Deák et al., 1997; Borchardt & Lienkaemper, 1999). 478 The diagenesis conditions at the soil surface support these micromorphological 479 features of calcite cement. It was also confirmed by lab experiments, which showed 480 that higher calcite supersaturation conditions tend to produce polycrystalline calcite 481

482 grains and corresponding aggregates (Mercedes-Martin et al., 2021). The 483 polycrystalline calcite mentioned above should have been formed in high calcite 484 supersaturation conditions, which were caused by high solar radiation, temperature 485 and aridity around the surface soil.

486 Short rods and rosette shapes means aragonite occurrence in the cement. Aragonite crystals usually show rods (Ma & Feng, 2015; Boyjoo et al., 2014; Cheng 487 et al., 2014; Sarkar & Mahapatra, 2012; Knez et al. 2006). Aragonite is more soluble 488 $(K_{sp}=6.3 \times 10^{-9})$ than calcite $(K_{sp}=4.0 \times 10^{-9})$ at ordinary pressures and temperatures 489 (25°C), and will commonly precipitate first from solutions. Aragonite is rare compared 490 491 to calcite because it is thermally unstable and will transform to calcite. This process is 492 more rapid in hydrous environments and at higher temperatures. For example, aragonite generally develops only under hot (e.g., springs) temperature conditions 493 (Zeng et al., 2018; Balthasar & Cusack, 2015; Gutjahr et al, 1996). At ordinary 494 temperatures the transition only takes place by dissolution of aragonite and 495 496 precipitation of calcite (e.g., Chen et al., 2003; Degens, 1965). Moreover, aragonite is 497 preferentially synthesized over calcite in the presence of (molten lauric) acid as an 498 additive (Sarkar & Mahapatra, 2012). This implies that presence of an organic acid is favourable for aragonite formation. Acid solution formation needs acid gases like CO₂, 499 H₂S, NO₂, etc. from decomposition of organic matter in aqueous soil. The decaying 500 Artemisia roots satisfy the acid environment formation in the desert soil (Sun et al., 501 2021, 2020). The rosette shape particles which were observed result from the 502 503 aggregation of aragonite rods. These characteristics were supported by the diagenesis features around the shallow soil of this desert. Hence, high temperatures, root 504 505 decomposition and possible high local Mg/Ca from weathering of soil particles in shallow soil favour aragonite formation. 506

507 The fact that calcified fungal hyphae networks were found in cement (Fig. 9b) in 508 the shallow soil or above the soil implies that soil microorganisms play a fundamental 509 role in the genesis of calcic soils around the rhizosphere. Biomineralization of calcite 510 by soil microorganisms has important implications for identifying subaerially exposed

511 sediments in the geological column (Wright, 1986). Calcified soil microorganisms, in addition, may serve as useful microfossils that reflect paleoclimatic conditions. The 512 mechanism explaining the production of calcite by microorganisms is probably 513 related to the exudation of excess Ca^{2+} by microorganisms, which concentrate Ca^{2+} on 514 their external surfaces (Phillips et al., 1987). In addition, the production of CO_2 by 515 microbial respiration contributes to the formation of bicarbonate ions (Arnott & 516 517 Pautard, 1970), which prompts calcite precipitation. Organic matter decomposition is increased by warm temperatures but is decreased by dryness. Overall, such a 518 decomposition is increased when the soil profile receives infrequent precipitation that 519 activates the dormant microbial population. High decomposition rates combined with 520 low carbon inputs explain the low amount of organic carbon in hot deserts (Khormali 521 & Monger, 2020). Calcified hair-like filaments are widely accepted to be of fungal 522 (hyphae) origin, but other micro-organisms such as algae, cyanobacteria, 523 actinomycetes, and eubacteria, and root hairs may also form these filaments (Eren et 524 525 al., 2018; Durand et al., 2010; Alonso-Zarza et al., 1998). The present results indicate 526 that calcified fungal filaments (Monger et al., 1991) can also be formed in desert soils that were initially noncalcareous, and whose calcite can be attributed to pedogenesis. 527

The relationships between diagenesis environment and micro-morphologies of 528 cementing minerals are summarized in Fig. 13. PRs are in the first step of diagenesis; 529 530 the vertical upper RRs above ground of the PRs are in the second step of diagenesis; the RRs lying on soil surface are in the last step of diagenesis. The 531 micro-morphologies of cementing minerals of PRs and RRs show different 532 characteristics related to the different diagenesis environments: (i) homogeneous 533 534 micritic carbonate mass with pores, cavities and voids formed at first diagenesis stage underground; (ii) short rods of aragonite formed at late diagenesis stage with higher 535 temperature; (iii) big and clear carbonate mineral crystals formed at last diagenesis 536 stage. Microorganisms are involved in the formation of calcified fungal hyphae 537 networks in favourable conditions at the dune soil surface, as confirmed by the 538

presence of desert biological soil crust (e.g., Zhang et al., 2021; Qi et al., 2021; Zhao
et al., 2020).

541

542 **6.** Conclusions

The characteristics of the cementing minerals of the carbonate rhizoliths in the Badain Jaran Desert are affected by (micro)environmental conditions in the dune sandy soil. These objects enable the assessment of past environmental conditions that prevailed during their formation.

547 Diagenesis environments are different between the deep and surficial dune soils in the desert. Within the deep soil, the moisture content is higher than at the surface 548 and the temperatures are stable. In contrast, the soil surface is characterized by large 549 day-night temperature amplitude coupled with strong wind deflation, erosion and 550 weathering under solar radiation. Thereafter, primary and reworked rhizoliths are 551 552 formed within and on the soil, respectively. The primary rhizoliths buried underground, the vertical reworked rhizoliths above soil, and the lying or horizontal 553 root tubes on soil surface correspond to early, middle and late diagenesis steps. The 554 555 calcium carbonate cement of the PRs buried underground mainly displayed homogeneous micritic mass calcite with pores, cavities and voids. In contrast, the 556 upper vertical parts-RRs of the PRs show short rods-like calcite crystals. However, 557 the lying or horizontal RRs displayed a large variety of morphology (polyhedral 558 559 crystals of calcite with well-developed crystal faces and slightly smooth edges and corners, or euhedral rhombic crystals, or prismatic/tooth-like crystals; rosette shapes 560 and short rods of aragonite; prismatic/pillar crystals; calcified fungal hyphae). This 561 confirms that the mineral crystalline morphologies are deeply affected by the different 562 diagenesis stages and are dependent on the nature of the soil environments. 563 Homogeneous micritic calcite crystals are formed during the initial stages of 564 diagenesis underground with a stable environment with wet soil, semi-closed and 565 -redox conditions. In contrast, the presence of polyhedral crystals of calcite with 566 well-developed crystal faces and slightly smooth edges and corners, or euhedral 567

rhombic crystals, or prismatic/tooth-like crystals as well as rosette shapes and short 568 569 rods of aragonite imply recrystallization through secondary diagenesis with complex 570 and long processes at the soil surface. Aragonite is preferentially formed at higher temperature and with higher concentration of mineral solution in shallow soil 571 environments. The presence of calcified fungal hyphae networks (hair-like, or 572 fungi-form) crystals and pores, cavities and voids somewhere among the micritic 573 574 mass indicate the role of microorganisms in calcium carbonate crystallization. The extreme environmental conditions and lack of water at the soil surface in hyper-arid 575 deserts hamper microbial life, allowing only highly specialized microbial 576 communities to grow and survive, specifically at the soil surface. 577

The micro-morphological characteristics of the rhizolith cement shows that they can be good proxies for soil formation, evolutions, pedologic and diagenesis processes, especially around and within rhizosphere in desert soil. This study provides a good reference and model for the rhizolith study of deserts worldwide.

582

583 **Declaration of competing interest**

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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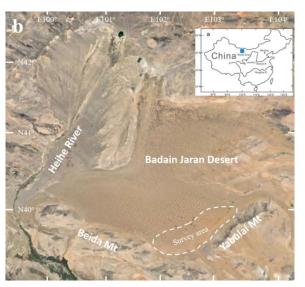
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886 Figures and Captions

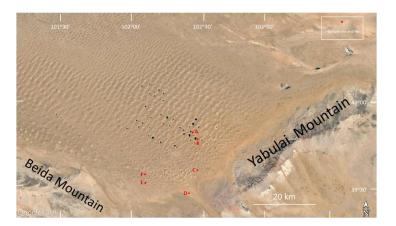
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- 889 Fig.1. Location (blue square) of the BJD in China (a) and the exploration area (white dash lines) in the
- 890 desert (b, Revised from https://earth.google.com).

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- 893 Fig.2. Detail of the sampling sites (A-F) in the southeastern desert. (A-Sumujilin Lake, B-Nuoertu Lake,
- 894 C-Baoritaolegai Lake, D-The Gate of National Geological Park of BJD, E & F-random sites among the
- dunes. Revised from https://earth.google.com)
- 896



898 Fig. 3. a-Fragments of caliche and rhizoliths (The spade length is 100 cm). b-Hard varnished

899 rhizoliths (The diameter of the coin is 20 mm).

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- 902 Fig. 4. White pieces of rhizoliths and a root tube. Relicts of fine root hairs within the tube and
- 903 weathered rills are observed on the surface of the tube.
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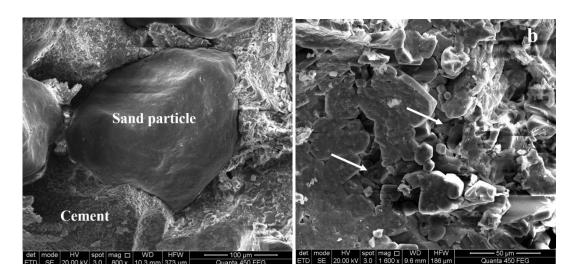
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- 909 Fig. 5. a-Vertical PRs dug out of soil (the red screwdriver handle length is 10 cm). b-Root relicts with
- 910 central cavity and beige peripheral part of PRs in Fig a.
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- 913 Fig. 6. Brown interior of the PR with root relicts.
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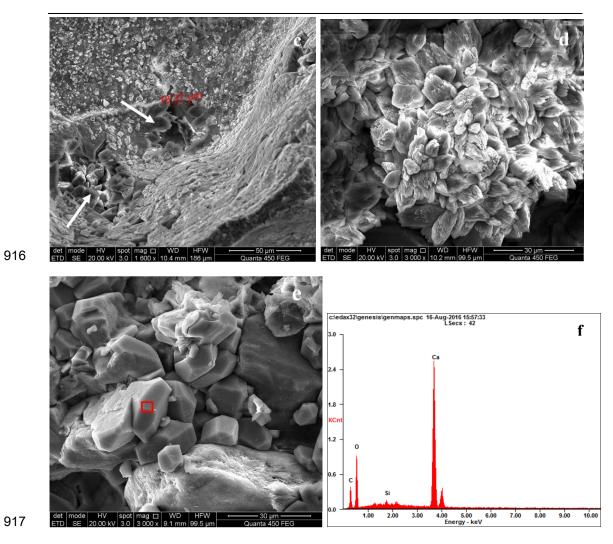
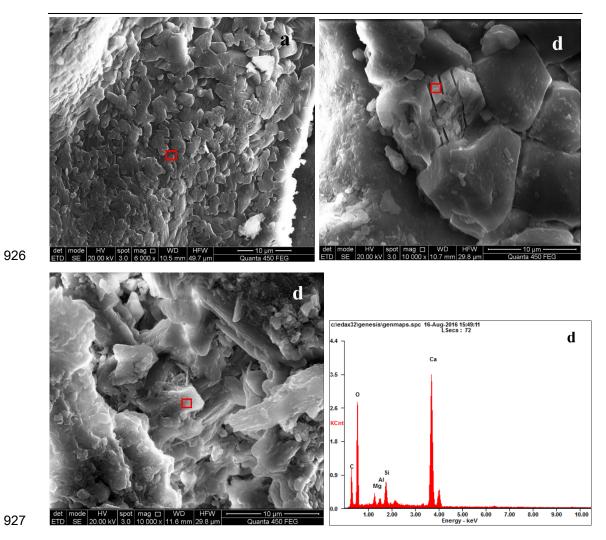
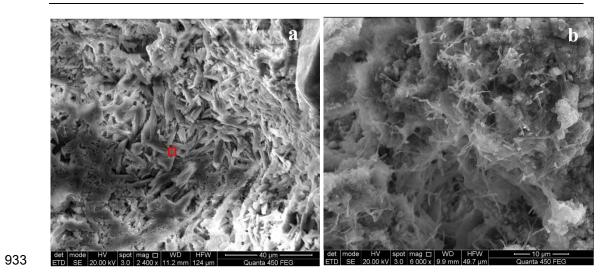


Fig.7. a-SEM microstructures of RRs. b-Polyhedral crystal cement and pores (white arrows). c-Some
of the cementing crystals are bigger euhedral rhombic crystals, showing prismatic/tooth-like shape
around the walls of the pores (white arrows). d-Aragonite rosette by morphology. e-Cement
polyhedral crystals, showing well-developed equant faces and slightly smooth edges and corners. The
crystals are modified scalenohedral or pentagonal repeated twins. The red square is the focus of the of
EDS analysis (same for the following Figs. 8-12). f-EDS spectrum reveals that the cement is made of
calcite (of Fig. e).



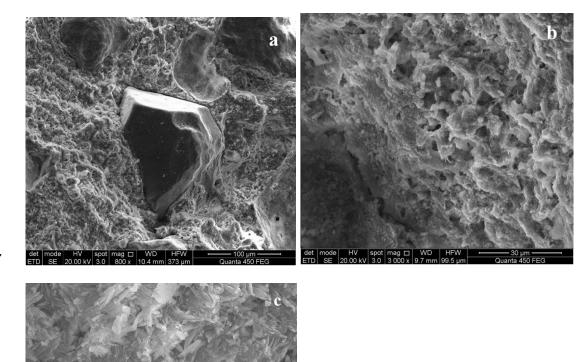
928 Fig.8. a-Spherular cementing minerals. b-Monocrystalline polyhedral calcites of cement. The EDS
929 results of Fig. a and b were omitted. c-Short rods. d-EDS spectrum (of Fig. c) reveals cement
930 calcium carbonate with a little portion of Mg.



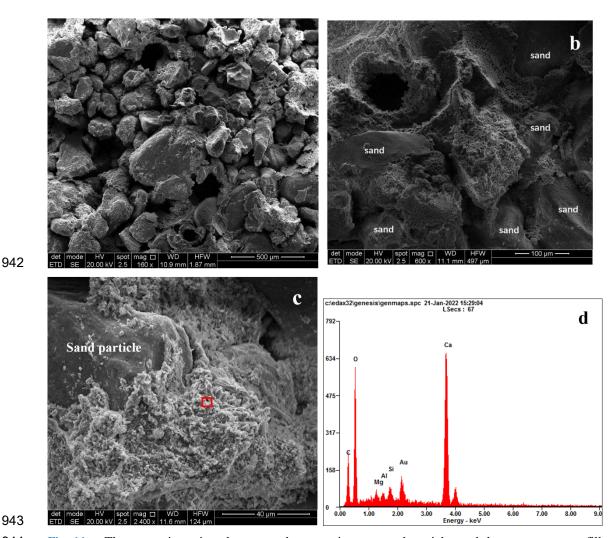
934 Fig.9. a-Short rods of aragonite. b-Hair-like, or fungi-form crystals of cement with pores, cavities

and voids.

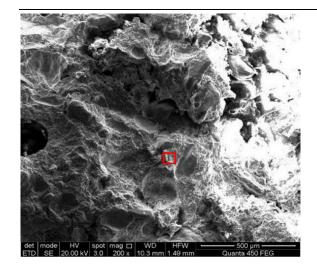
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- 939 Fig.10. a-Calcite cement around a sand particle, showing homogeneous micritic mass (PR). b-Pores,
- 940 cavities and voids among the micritic mass (PR). c-Dense cement of prism/pillar-like calcite (PR).
- 941



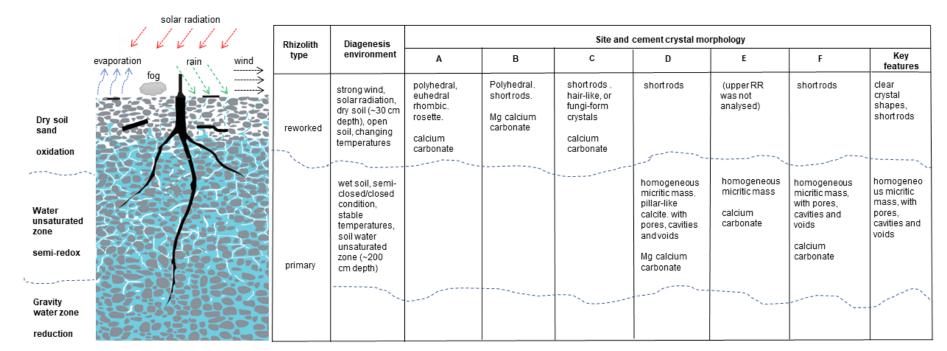
944 Fig. 11. a-The cementing minerals are mostly as coatings on sand particles and the cement cannot fill 945 and plug the pores among sand particles. Therefore, large pore spaces among the particles remain 946 empty and the particles are loose because of a porous cement. b-The cementing minerals are 947 homogeneous micritic mass. c-The homogeneous micritic mass is forming thin layers on the surfaces 948 of particles. d-EDS spectrum (of c) reveals that the cement is mainly constituted of calcium carbonate 949 with only a little Mg.



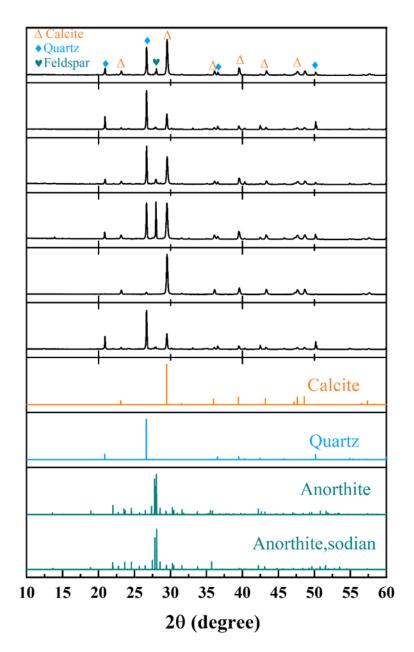
- 951 Fig. 12. Homogeneous micritic mass.

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⁹⁶⁰ Fig. 13. Rhizolith types, diagenesis environment, micro-morphologies and compositions of cementing minerals at different sites.



966 Fig. 14. XRD patterns of the cements collected at different sites: a-Site A, b-Site B, c-Site C, d-Site D,
967 e-Site E, f-Site F. The standard card numbers of calcite, quartz, and anorthite used here are #86-2334,
968 #85-0797 and #10-0393, respectively.